

PATENT ABSTRACTS OF JAPAN

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(71)Applicant : NITTO KASEI CO LTD

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(72)Inventor : OKADA TAKAYUKI
NAKAMURA HIROMASA
TABUCHI HITOSHI
MORI KIYOMI

(54) MOISTURE-CURABLE COMPOSITION

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a modified silicone moisture-curable composition which has a high curing rate and uses a catalyst system having a low toxicity.

SOLUTION: The moisture-curable composition contains a mixed catalyst comprising from 0.1 to 10 pts.wt. bismuth compound (B) and a reaction product obtained by allowing, based on 1 mol bismuth compound (B), from 1 to 7 mol, calculated as the molar number of carboxyl groups, compound (C) having at least one carboxyl group to react with from 0.6 to 3.5 mol, calculated as the molar number of amino groups, compound (D) having at least one amino group as a curing catalyst against 100 pts.wt. silyl group-containing organic polymer (A) having at least one hydrolyzable group-bound silicon atom per molecule at the molecular terminal or the side chain.

* NOTICES *

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1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. *** shows the word which can not be translated.

3. In the drawings, any words are not translated.

CLAIMS

[Claim 1a]

(Claim 1) As opposed to silyl group content organicity polymer (A) 100 weight section which has the silicon atom combined with a molecular terminal or a side chain with a hydrolytic basis in [at least one] one molecule, as a curing catalyst — the biemuth compound (B) 0.1 — ten weight sections and based on quantity of 1 mol of a biemuth compound (B), A carboxyl group, A compound (C) which has at least one amino group, and a type compound containing a mixed chain which consists of 0.5-3.5 mol of one piece. And based on quantity of 1 mol of a carboxyl group, A compound (D) which has at least one amino group, and a type compound containing a mixed chain which consists of 0.5-3.5 mol of one piece. And based on quantity of 1 mol of a carboxyl group, A compound (D) which has at least one amino group based on the number of moles of a carboxyl group.

(Claim 2) A biemuth compound (B) is a general formula (1).

$B(OOCR^1)_3$ (1)

The wet cure type composition according to claim 1 which is biemuth carboxylate expressed with (inside of formula and R^1 is a hydrocarbon group of the carbon numbers 1-17).

(Claim 3) The wet cure type composition according to claim 1 or 2 whose biemuth compound (B) is biemuth tris (2-ethylhexanoate) or biemuth tris (neo decanoate).

(Claim 4) The wet cure type composition according to any one of claims 1 to 3 whose compound (C) which has at least one carboxyl group is straight chain shape or branched-chain aliphatic carboxylic acid of saturation of the carbon numbers 2-15, or an unsaturation.

(Claim 5) The wet cure type composition according to any one of claims 1 to 4 whose compound (D) which has at least one amino group is primary amine.

(Claim 6) A catalyst is a biemuth compound (B) based on quantity of 1 mol of a biemuth compound (B). A catalyst is a biemuth compound (B) based on quantity of 1-3 mol based on the number of moles of an amino group in compound (D) which has 2-5 mol and at least one amino group for a compound (C) which has at least one carboxyl group based on the number of moles of a carboxyl group is blended. The becoming wet cure type composition according to any one of claims 1 to 5.

[Translation done.]

restrictions in particular, the thing of polymers is hyperviscosity, and since it becomes difficult [a use top] when it is considered as a hardening setup-of-tooling product, 30000 or less are too desirable [a thing] as a number average molecular weight. Although such an organic polymer can be manufactured by a pulvisio known method, commercial items, such as KANEKA MS polymer by Kaneka Corp., may be used for it.

[0017] As a biimide compound (B) used for this invention, one sort of the biimide SUTORI carboxylic acid expressed with said general formula (1) or two sorts or more are used preferably. As carboxylic acids of the carbon numbers 2–18 which give the carboxyl group shown by OCO(R1) in a general formula (1), Acetic acid, propionic acid, butanoic acid, isobutyric acid, pentanoic acid, hexanoic acid, heptanoic acid, Octanoic acid, 2-ethylhexanoic acid, nonanoic acid, decanoic acid, undecanoic acid, dodecanoic acid ($\text{C}_{12}\text{H}_{25}\text{O}_2$), tridecanoic acid, tetradecanoic acid, pentadecanoic acid, hexadecanoic acid ($\text{C}_{16}\text{H}_{33}\text{O}_2$), heptadecanoic acid, octadecanoic acid, stearic acid, myristic acid, palmitic acid, and oleic acid, or an unsaturation is raised. If the biimide SUTORI carboxylic acid expressed with a general formula (1) is illustrated concretely, Biimide triacetate, biimide tripalmitate, biimide SUTORI heptanoate, Screw melt ROKUMETO, biimide tri-(2-ethylhexanoate), biimide tri-(oleo decaoctate), biimide trimaleate, a biimide trioctate, biimide triheptanoate, etc. are raised. Biimide bis-(2-ethylhexanoate) and biimide tri-(non decaoctate) are [among these] preferred. Such a compound can be manufactured by a publicly known method.

[illegible]

[2019] As a series (C) which has at least one amino group and calling it transfer amino group containing compound (DD), primary amine is used preferably. When it illustrates concretely, as primary amine methylamine, ethylamine, propylamine, isopropylamine, isopropyl alcohol amine, *n*-butylamine, 1-ethyl butylamine, isobutyl amine, pentylamine. To two, PUCHIERI amine, octylamine, two decyl amine, lauryl amine. Monocyclamine, dicyclamine propylamine, dicyclamine, cyclohexylamine, benzylamine, guanidine, 2-ethylhexylamine, Aliphatic series primary amine, such as triethylenetriamine, aniline, a nitroaniline. Aromatic series primary amine, such as a phenylendamine, a toluidine, tolyl amine, xyloendamine, a 2,3-bisphenol, an aniline, phenetidine, benzidine, benzylamine, and naphthylamine, is related. Cycloamine, two decyl amine, lauryl amine, 1-ethylhexylamine, and benzylamine are preferred, and tolyl amine and especially 2-ethylhexylamine are preferred.

[0020] Before adding to a silyl group containing an amine polymer (A), it is necessary to make a carboxylic compound (C) and an amine group out of this compound. If a carboxylic compound (C) is added to an amine group (A) without a carboxylic compound (C) and an amine group out of this compound, it may not occur and a sufficient reaction may not occur. A carboxylic compound (C) may be made to add and react to a reactant (A). An amine group containing compound (D) may be made to add and react to a carboxylic compound (C), and a carboxylic compound (D) may be made to add and react to an amine group containing compound (C). A reaction is performed usually stirring both mixed liquid (when a mixture is not liquefied, it heats, and it is made liquefied or a suitable solvent is added) under the absence of a solvent. A reaction must be thoroughly performed until reaction fever stops occurring.

[0021]The mixed catalyst used for this invention may add and prepare a biamuch compound (B) to the reactant of the above-mentioned carboxyl containing compound (C) and an amino group containing compound (D). An amino group containing compound (D) is added into the mixture of a biamuch compound (B) and a carboxyl containing compound (C), and under existence of a biamuch compound (B), a carboxyl containing compound (C) and an amino group containing compound (D) may be made to react, and it may prepare. A carboxyl containing compound (C) is added into the mixture of a

bismuth compound (B) and an amino group containing compound (D), and under existence of a bismuth compound (B), a carboxyl containing compound (C) and an amino group containing compound (D) may be made to react, and it may prepare. A bismuth compound (B), a carboxyl containing compound (C), and an amino group containing compound (D) are added at a time, and under existence of a bismuth compound (B), a carboxyl containing compound (C) and an amino group containing compound (D) may be made to react, and it may prepare.

[0222] In the present invention, the content of a biarnth compound (C) is preferably 0.1 to 10 preferred weight, based on 100 weight of a biarnth compound (B). The content of a biarnth compound (C) is 0.1 to 10 preferred weight, based on 100 preferred weight of a biarnth compound (B). When said less than range of hardening performance is observed, the content of a biarnth compound (C) is insufficient and it exceeds said range on the other hand, physical properties, such as recovery of the hardened material after hardening and weatherability, may worsen. The content of a carboxyl containing compound (C). Based on the number of moles of a carboxyl group, the quantity of 1 mol of a biarnth compound (B), 2-5 mol, preferably, 3 mol most preferably more preferably 1-7 mol is preferred and 1, and 3-4 mol especially the content of an amino group containing compound (C), 2-3 mol-1.3 mol is 2 mol most preferably especially preferably more preferably 0.6-3.5 mol is preferred to the quantity of 1 mol of a biarnth compound (B), and 1 if said less than range of hardening performance is [the content of a carboxyl containing compound (C)] insufficient and it exceeds said range on the other hand, hardening performance degradation will be seen. If said less than range of hardening performance is [the content of an amino group containing compound (C)] insufficient and it exceeds said range on the other hand, hardening performance degradation will be seen.

[0023] In order to promote hardening in the moisture curing nature constituent of this invention and to improve adhesion to a substrate, can use publicly known various amino group substitution alkoxy silane compounds or the condensate of those, and specifically, gamma-aminopropyltrimethoxysilane, gamma-aminopropyl triethoxysilane, N-(trimethoxysilyl)propyltriethylenediamine, delta-aminobutyl (methyl) diethoxysilane, N, and N'-bis(trimethoxysilyl)propyltriethylenediamine, delta-aminobutyltriethoxysilane, etc. are mixed.

ethyleneamines, these partial hydroxylates, etc. are raised. [0024] The additive agent usually further added by its availability. For example, a hardener accelerator, a concrete retarder, a dipping inhibitor, an antiaging agent, and a solvent, may be added to the moisture curing nature of this invention. As a bulking agent, specifically for example, calcium carbonate, kaolin, talc, Fumed silica, sedimentation nature silica, a silicic acid hydroly, hydrous silicic acids, talc, Colloidal silica, bentonite, organic bentonite, a mill ballon, glass fibers, asbestos, a glass filament, granular diatomite, aluminum silicate, aluminum hydroxide, a zinc oxide, magnesium oxide, a titanium dioxide, etc. are raised. Specifically as colorant, iron oxide, carbon black, copper phthalocyanine blue, Phthalocyanine Green, etc. are used. As a plasticizer, specifically Dibutyl phthalate, dioctyl phthalate, Dioctyl sebacate, butyl benzyl phthalate, dioctyl phthalate, Siccinic acid didecyl, succinic acid diundecyl, diisododecyl phthalate, dioctyl phthalate, Siccinic acid didecyl, succinic acid diundecyl, diisododecyl phthalate, Dioctyl ester, such as aliphatic-carboxylic-acid ester species, such as butyl oleate, and pentaerythritol ester. Epoxy plasticizers, such as phosphoric ester, such as triethyl phosphate and triethyl phosphite, azodized soybean oil, and epoxy terearic acid benzyl, a chlorinated paraffin, etc. are used. Specifically as a dipping inhibitor, hydrogenation ostar oil, a silicic acid hydroly, organic bentonite, colloidal silica, etc. are used. As other additive agents, adhesion grant agents, such as phenol resin and an epoxy resin, an ultraviolet ray absorbent, radical chain inhibitor, a peroxide decomposer agent, various kinds of antiaging agents, etc. as used.

[0025] [Example] although this invention is concretely explained based on an example below, the range of this invention be elaborate --- be alike --- *** is not limited.

000226 In the attached 200-ml eggplant type flask, an example of manufacture 1 nitrogen introducing pipe 43.3 g (0.3 mol) of 2-ethylhexanoic acid, 37.0 g (0.2 mol) of lauryl amine and 63.9 g (0.1 mol) of bismuth tris (2-ethylhexanoate) were weighed, and it fully mixed with the magnetic stirrer. Generation of heat was subsided, stirring was continued until the internal temperature became near the room temperature, and the fluid of yellow transference was obtained.

[0027] In the attached 200-ml eggplant type flask, an example of manufacture 2 nitrogen introducing compound, and the fluid or yellow a single structure was added.

